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Preliminary report on the clay mineralogy of the Upper Devonian shales in the southern and middle Appalachian basin

by

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Abstract

The distribution of kaolinite in parts of the Devonian shale section is the most significant finding of this work. These shales are composed predominately of 2M illite and illitic mixed-layer clay with minor amounts of chlorite and kaolinite. Preliminary data indicate that kaolinite, the only allogenic clay mineral, is present in successively older beds of the Ohio Shale from south to north in the southern and middle parts of the Appalachian basin. This trend in the distribution of kaolinite shows a paleocurrent direction to the southwest.

Three well-known methods of preparing the clay fraction for X-ray diffraction analysis were tested and evaluated. Kaolinite was not identified in two of the methods because of layering due to differing settling rates of the clay minerals. It is suggested that if one of the two settling methods of sample preparation is used, the clay film be thin enough for the X-ray beam to penetrate the entire thickness of clay.

Introduction

A total of 441 samples of Devonian shale have been analyzed for their clay content. The samples were obtained from 9 cores drilled recently for the Eastern Gas Shales Project of the Department of Energy and from 8 cores drilled during 1948 to 1952 for the uranium program of the Atomic Energy Commission. Samples were taken at 5, 10, 20, or 25-foot intervals. The number of samples per drill hole ranges from 3 to 77 depending upon the length of core and sampling interval. The following table gives the API (American Petroleum Institute) identification number for each drill hole, (Fig. 1) approximate location by

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Figure 1 near here.

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county and state, the interval cored in each drill hole, and the number of samples analyzed from each core. This report was prepared in cooperation with the Department of Energy under interagency agreement EX-76-C-01-2287.

Large sections of many of the drill holes were not cored and samples are not available. There is also a discrepancy between the wire logs and footage marking on the cores. No attempt was ever made to establish the amount of this discrepancy even though it was known to exist. To obtain information of the regional sedimentary pattern, more sampling of shale sections are needed throughout the central and northern parts of the Appalachian basin.

Figure 1.-- Index map showing location of drill holes.

API number	Location	No. of samples	Core interval (feet)
01-009-0000	Blount Co., Ala. 2 miles northeast of Blountsville	5	16.4-49.7
41-031-0000	Coffee Co., Tenn. Road intersection at Hoodoo	7	85.9-116.8
41-035-0000	Cumberland Co., Tenn. 10 miles south of Crossville	8	139.3-174.2
41-037-0000	Davidson Co., Tenn. Caney Creek valley, 1.3 miles south of U.S. 41A	5	19.9-40.0
41-041-0000	Dekalb Co., Tenn. 3.1 miles northwest of Smithville	9	138.6-173.1
41-115-0000	Marion Co., Tenn. 1 mile south of Kelly Chapel	3	293.8-307.6
41-127-0000	Moore Co., Tenn. 0.5 mile north of Cumberland Springs dam	4	8.8-21.1
41-165-0000	Sumner Co., Tenn. 1.5 miles west of Camp Creek on Long Hollow Pike	5	14.1-37.0
16-159-31029	Martin Co., Ky.	49	2429-3411
16-195-28982	Perry Co., Ky. 12 miles north of Hazard	34	2369-2708
34-019-20835	Carroll Co., O. 12 miles northwest of Carrollton	25	2080-2200 3080-3200
34-167-23521	Washington Co., O.	25	3490-3714
47-035-21369	Jackson Co., W. Va. 5 miles southwest of Cottageville	26	3410-3500 3600-3794

API number	Location	No. of samples	Core interval (feet)
47-035-21371	Jackson Co., W. Va. 3 miles west of Cottageville	47	3220-3690 4020-4030
47-043-21636	Lincoln Co., W. Va. 3 miles southwest of Ranger on Ten Mile Creek	59	2654-2770 3000-3118 3886-3968
47-043-21637	Lincoln Co., W. Va. 3 miles southwest of Ranger on Fourteen Mile Creek	53	2720-4028
45-035-21369	Wise Co., Va.	77	4870-4986 5210-5475

Investigations of the mineralogy were based primarily on X-ray diffraction analyses of the whole sample and the clay fraction of each sample. For this report the term "clay fraction" is defined as the hydrous aluminum silicates or clay minerals and is not used as a term for particle size. The reason for this is because the Devonian shales and siltstones are highly indurated and cannot be disaggregated without grinding which probably reduces some of the silt-size grains to clay-size particles. For identification of the clay minerals, the normal procedure was used for separating to clay fraction.

Sample preparation

Each sample was ground in alcohol in a tungsten-carbide ball mill for 15 minutes. To obtain X-ray analysis of the bulk rock, a small portion of the ground material was further ground by hand to pass through a 200-mesh screen and then pressed into a 29-millimeter diameter wafer which was analyzed to obtain an X-ray diffraction trace of the whole sample. To properly identify the clay minerals in each sample, the remainder of the ground material was dispersed in deionized water. No chemicals were added to this clay-water mixture unless the sample could not be deflocculated. If flocculation occurred, an ultrasonic horn was placed in the clay-water mixture for about 15-30 minutes. Usually this treatment stopped the flocculation, but if it did not, then a few drops of sodium metaphosphate solution (0.05 g of $NaPO_3$ dissolved in 1 cc of water) was added to assure dispersal of the clay minerals. The clay fraction obtained by settling was siphoned off and placed on a porous ceramic tile using the vacuum technique described by Kinter and Diamond (1956).

Samples were analyzed on a standard X-ray diffraction unit using copper ($CuK_{\alpha 1}$) radiation in conjunction with the following optical and recording arrangements: A 1° beam slit, a medium resolution soller slit, a 0.1° detector slit, a graphite crystal for monochromatic radiation, and a 5° take-off angle. The goniometer was run at a speed of 1° 2θ per minute and the chart recorder was driven at a speed of 12 inches per hour, so that the X-ray diffraction trace was recorded at a rate of 5° 2θ per inch. The detector was set on a linear scale of 1000 counts per second (cps) for sull-scale deflection and a 2.5 second time constant was used. The X-ray diffraction trace was used to identify and determine relative abundance of each clay mineral following the method of Schultz (1960). An electronic planimeter was used to carefully measure selected peak areas on the X-ray diffraction traces to obtain the relative proportions of the individual clay minerals. X-ray diffraction traces of the whole sample will be used to determine the bulk mineralogy.

It is important to note that different sample-preparation techniques may fail to identify all clay minerals present in the original sample. Therefore, we tested and evaluated three well-known methods of preparing the clay fraction for X-ray diffraction analysis. Method A settles the clay fraction onto a glass slide by using a centrifuge. Method B settles the clay fraction onto a glass slide by allowing the water to evaporate from the clay-water mixture. Method C places the clay-water mixture onto a porous ceramic tile and removes the water through the tile by vacuum (Kinter and Diamond, 1956). X-ray diffraction traces were made on the top surface of all three preparations, and X-ray diffraction traces were made of the bottom surface (the surface in contact with the glass slide) of the first two preparations. An X-ray diffraction trace of the bottom surface of the sample prepared on a porous tile by vacuum could not be made because the sample could not be removed from the tile. The results are shown on figure 2. The

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Figure 2 near here.

X-ray diffraction traces of the top surfaces of two sample preparations A and B, fig. 2)indicate a single peak at 25° 2θ and the intensity of the 9° 2θ peak is much larger than the 12.5° 2θ peak. Quartz, represented by the 20.8° 2θ peak, seems to be present in very minute amounts on the surface. The X-ray diffraction traces of the bottom surface (D and E, fig. 2) and the X-ray diffraction trace using the vacuum method (C, fig. 2) indicate the presence of both kaolinite and chlorite

Figure 2.--X-ray diffraction traces of a sample prepared three different well-known ways. The sample is from drill hole (API #47-035-21369) Jackson Co., West Virginia, at a depth of 3489.0 feet. The vertical scale is 200 counts per second except A, which is 500 counts per second.

A. X-ray diffraction trace of top surface of settled sample, water removed by centrifuge.

B. X-ray diffraction trace of top surface settled sample, water removed by evaporation.

C. X-ray diffraction trace of top surface of sample on porous ceramic tile, water removed by vacuum.

D. X-ray diffraction trace of bottom surface of A.

E. X-ray diffraction trace of bottom surface of B.

by the double peak at 25° 2θ; also, the intensities of the 9° 2θ peak and the 12.5° 2θ peak in all three traces are more nearly equal. Quartz is more abundant on the bottom surface (D, and E, fig. 2) than on the top surface as indicated by the 20.8° 2θ peak. This would strongly suggest the possibility that there is layering due to differing rates of sedimentation when the $<2 \mu m$ fraction is allowed to settle either by centrifuge (Method A) or by evaporation (Method B). Another obvious effect of layering is the double peak at 25° 2θ which indicates the presence of the (002) peak for kaolinite and the (004) peak, of chlorite. Kaolinite, which is usually coarser-grained than either illite or chlorite, is the first to settle and therefore does not show on the X-ray traces of the top surface. The vacuum method of Kinter and Diamond (1956, p. 111) minimizes or eliminates this layering effect which may be present when the sample is allowed to settle.

We favor the vacuum method of sample preparation and have used it in the identification of clay minerals for this report. We suggest that if either settling method of sample preparations is used, the clay fraction on the glass-slide be thin enough for the X-ray beam to penetrate the entire thickness of sample.

Clay mineralogy

The clay minerals found in Devonian shale of the Appalachian basin are 2M illite (muscovite), chlorite, and kaolinite. Considerable illite and some mixed-layer clay was found in all samples. Small quantities of chlorite are found in all but one or two samples, and very small amounts of kaolinite were present in about 30 percent of the samples. The proportions of illite and mixed-layer clays were determined by measuring the changes in the area of the 10A (9° 2θ) peak by using a planimeter. Proportions of chlorite and kaolinite were determined from a ratio obtained by measuring the areas of the double peak at about 3.5A (25° 2θ), and applying that ratio to the 7A (12.5° 2θ) peak.

The term "illite" $(OH)_4K_2(Si_6Al_2)(MgFe)_6O_{20}$ in this report is applied to a dioctohedral clay-size 2M polytype of muscovite. It contains 12 percent K_2O, 39 percent Al_2O_3, 45 percent SiO_2, and 5 percent H_2O. Illite is the most abundant clay mineral with relative proportion ranges from 40 to 90 percent. It is recognized on X-ray diffraction traces by its strong first order basal (001) peak at approximately 10A, a weak second-order basal (002) reflection at 5A, and a strong third-order basal (003) reflection at 3.3A. The 2M polymorph is identified by the position of the non-basal reflections that occur between 4.5A and 2.5A. Only the material that does not expand at the 10A position when treated with ethylene glycol is considered to be illits; the expandable material is considered to be mixed layer clay.

Mixed-layer clays are the random interlayering of two or more clay minerals, and illite is commonly one of the major constituents. Two types of mixed-layer minerals were identified in samples of Devonian shale: an expandable type and a non-expandable type. The expandable type is composed of a random mixture of illite and an expandable clay mineral such as smectite. The non-expandable type is composed of a random mixture of illite and either degraded chlorite or vermiculite. The mixed-layer minerals are identified by comparing measurements of the 10A peak area on X-ray diffraction traces of the clay fraction that have been run untreated, run after saturation with ethylene glycol, and run after heating at 350°C for a minimum of 30 minutes. Saturation with ethylene glycol expands the montmorillonite layers to a higher spacing leaving pure illite in an amount represented by the 10A peak area. Therefore, the 10A peak area on the trace of an untreated sample represents pure illite plus the expandable type mixed-layer. Heating the sample collapses the non-expandable mixed layer to the 10A position; therefore, the 10A peak area on the trace of the heated sample represents illite and both types of mixed-layer clay. From the measurements of these three areas, an estimate can be made of the amount of illite, expandable mixed-layer, and non-expandable mixed-layer clay in each sample.

Chlorite $(OH)_8(SiAl)_4(MgFe)_3(MgAl)_3O_{10}$ was present in almost all samples of Devonian black shale analyzed for this report. It contains 14 percent MgO, 27 percent Fe_2O_3, 17 percent Al_2O_3, 30 percent SiO_2, and 12 percent H_2O. The relative proportion of chlorite ranged from a trace in a few samples to 30 percent in a few samples with the average sample containing between 10 and 15 percent. Chlorite is recognized on X-ray diffraction traces by its moderate to weak first-order basal (001) peak at 14A, its strong second-order basal (002) reflection at 7A, its peak third-order basal (003) reflection at about 4.7A, and its weak to moderate fourth-order basal (004) peak at approximately 3.5A. Chlorite is not affected by ethylene glycol saturation or heating to 350°C, however, produces an increase in the first-order basal (001) peak at 14A and increases in the second, third, and fourth-order basal reflections. Fine-grained chlorite decomposes in warm hydrochloric acid (Brindley, 1961, p. 264) but it may be necessary to keep the temperature at about 80°C for 16 to 20 hours to achieve complete decomposition, especially if the chlorite is well crystalline.

Kaolinite $(OH)_8Si_4Al_4O_{10}$ is the least abundant clay mineral in the Devonian black shales that were analyzed. It contains 40 percent Al_2O_3, 46 percent SiO_2, and 14 percent H_2O. It occurred in about 30 percent of the samples in relative proportions that ranged to a maximum of 20 percent. Kaolinite is recognized on X-ray diffraction traces by its strong basal (001) peak at 7A and its moderate basal (002) reflection at approximately 3.5A. Kaolinite is not effected by ethylene glycol saturation or heating to 350°C for a minimum of 30 minutes. Heating kaolinite to about 550-600°C destroys its crystallinity; however, the exact temperature at which kaolinite is destroyed depends upon its grain size and crystallinity. Kaolinite is more immune to attack by warm dilute acids than most other clay minerals including chlorite; however, dissolution rates vary considerably with grain size and crystallinity.

It is very difficult to determine the presence of small amounts of kaolinite when chlorite is present. However, a slight difference in the c-axis dimension of the two minerals often are expressed as a doublet at 3.50A for the second-order basal peak (002) of kaolinite and the 3.57A for the fourth-order basal peak (004) of chlorite. We depended upon this doublet (at about 25° 2θ) for recognizing the existence of kaolinite. Heating to temperatures that destroy kaolinite also reduce the 7A and 3.5A peaks of chlorite. The acid treatment is fairly reliable for identifying kaolinite and we used this method as a check.

Non-clay minrals

The material other than the clay minerals include both allogenic minerals that were introduced during deposition of the clay and authigenic minerals that were formed at the time or shortly after the clay was deposited. The allogenic minerals are quartz, K-feldspar and plagioclase. The authigenic minerals are calcite, dolomite, siderite, pyrite, and barite. Organic matter is the only non-clay material that is probably both allogenic and authigenic. Quartz is present in all samples and pyrite is present in many samples, especially those high in organic matter. The feldspar seem to be associated with kaolinite. The other non-clay minerals occur in very minor amounts and only in a very few samples. Percentage of non-clay minerals in the samples is not available for this report.

Kaolinite distribution

Some very preliminary conclusions can be drawn from the clay mineral analysis of samples of Upper Devonian shale from 17 drill holes in the southern and middle parts of the Appalachian basin. The distribution of kaolinite, the only allogenic clay mineral, even in minor amounts, can indicate the direction of sedimentary transport because it is usually coarser-grained than the other clay minerals and in a marine environment, kaolinite is deposited near shore (Parham, 1966, fig. 6). The absence of kaolinite in the southern part of the basin suggests either the shales were deposited at a considerable distance from the shoreline or the 2M illite is the alteration product of kaolinite that was deposited in the original sediment. The former interpretation is favored by Harvey and others (1977) and we agree with this conclusion because of the complicated geochemical process required to convert kaolinite to 2M illite, the absence of authigenic silicate, and the fineness of illite. The illite was probably originally deposited as a Md polytype or degraded variety which normally results from the katamorphic alteration and erosion of crystalline and older sedimentary rocks. Diagenesis and low-grade regional metamorphism converted the Md illite to 2M illite. Velde (1965) found that 2M illite can become stable at a temperature as low as 125°C at 4.5 kb. This data agrees with Harris (1977) who compared conodont color alteration with degree of metamorphism in the Appalachian basin.

Tables 1 through 5 give the clay-mineral ratios for the 441 samples analyzed and their stratigraphic designation (Kepferle and others, 1977; Oliver and others, 1969; Roen and others, 1977a, 1977b; Wallace and others, 1977a, 1977b; and West, 1977). The following discussion outlines the presence of kaolinite in a south to north direction. Kaolinite was not found in the Chattanooga Formation of Alabama and Tennessee or in the Ohio Shale of Perry County, Kentucky. In Wise County, Virginia, kaolinite was found in the upper part of the Ohio shale in Martin County, Kentucky, and in Lincoln County and Jackson County, West Virginia, kaolinite was found in the upper and middle parts of the Ohio Shale. Kaolinite was found in the lower part Ohio Shale in Washington and Ohio Counties, Ohio. Kaolinite was not found in the Java or West Falls Formations which underlie the Ohio Shale in eastern Ohio and West Virginia. In summary, preliminary data shows that kaolinite is present in successively older beds of the Ohio Shale from south to north. This trend in distribution of kaolinite supports the paleocurrent data presented by Lundegard and others (1977) which shows a current direction to the southwest in the southern and middle part of the Appalachian basin, and is in keeping with a westward-prograding shoreline in the Late Devonian. Possible source areas for the kaolinite include the Canadian Shield and the Adirondack Mountains.

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Table 1. Clay mineral ratios of Devonian shale samples from selected drill holes in Alabama and Tennessee

Clay Mineral Ratio

Stratigraphic Unit	Footage	Chlorite	Illite	Swelling Mixed-layer	Non-swelling Mixed-layer	Kaolinite	color
API No. 01-009-00000			Blount Co., Alabama				
Chattanooge Shale-Gassaway Member	22.6	--	80	20	--	--	N2
" " " "	29.3	15	65	20	--	--	N2
" " " "	36.1	15	65	20	--	--	N2
" " " "	42.9	15	65	20	--	--	N2
" " " "	49.7	5	65	30	--	--	N2
API No. 41-031-00000			Coffee Co., Tennessee				
Chattanooga Shale-Gassaway Member	87.6	5	65	30	--	--	10YR3/.8
" " " "	92.5	10	55	20	15	--	10YR3/.9
" " " "	97.5	10	60	15	15	--	10YR3/.9
" " Dowelltown Member	102.5	10	70	15	5	--	10YR6/.2
" " " "	107.0	10	65	20	5	--	10YR6/.8
" " " "	112.0	10	65	15	10	--	10YR4/.5
" " " "	116.5	10	65	20	5	--	10YR4/.5
API No. 41-035-00000			Cumberland Co., Tennessee				
Chattanooga Shale-Gassaway Member	139.4	--	70	15	15	--	10YR3/.3
" " " "	144.5	5	65	20	10	--	10YR3/.3
" " " "	149.5	5	55	30	10	--	10YR3/.4
" " " "	154.5	5	65	20	10	--	10YR3/.3
" " " "	159.5	10	65	25	--	--	10YR3/.3
" " Dowelltown Member	164.5	15	70	15	--	--	N6
" " " "	169.5	15	75	10	--	--	N6.5
" " " "	174.1	--	90	10	--	--	N2
API No. 41-037-00000			Davidson Co., Tennessee				
Chattanooga Shale-Gassaway Member	14.9	10	60	30	--	--	10YR4/.9
" " " "	20.5	10	70	20	--	--	10YR4/.1
" " " "	26.5	10	70	20	--	--	10YR4/.9
" " Dowelltown Member	33.0	10	70	20	--	--	10YR5/.8
" " " "	40.0	10	70	20	--	--	10YR4/.7
API No. 41-041-00000			Dekalb Co., Tennessee				
Chattanooga Shale-Gassaway Member	142.4	10	75	15	--	--	N3
" " " "	147.4	10	80	10	--	--	10YR3/.5
" " " "	152.4	10	75	15	--	--	10YR3/.5
" " " "	157.4	10	70	20	--	--	10YR3/.5
" " Dowelltown Member	157.9	10	65	25	--	--	10YR4/.5
" " " "	163.0	10	80	10	--	--	10YR4/.3
" " " "	167.0	15	70	15	--	--	10YR3/.5
" " " "	168.0	10	70	20	--	--	10YR3/.8
" " " "	173.0	5	70	25	--	--	10YR3/.4
API No. 41-115-00000			Marion Co., Tennessee				
Chattanooga Shale-Gassaway Member	294.0	--	70	15	15	--	N2.5
" " " "	299.0	--	60	30	10	--	10YR3/.3
" " " "	304.0	--	65	30	5	--	10YR3/.3
API No. 41-127-00000			Moore Co., Tennessee				
Chattanooga Shale-Gassaway Member	8.8	10	85	5	tr	--	10YR3/.5
" " " "	12.6	10	60	30	--	--	10YR3/.5
" " Dowelltown Member	15.6	--	75	25	--	--	10YR5/.5
" " " "	20.2	10	70	20	--	--	10YR3/.5
API No. 41-165-00000			Summer Co., Tennessee				
Chattanooga Shale-Gassaway Member	14.2	5	85	10	--	--	10YR3/.5
" " " "	20.0	10	70	20	--	--	10YR4/.8
" " " "	23.8	10	60	30	--	--	10YR4/.9
" " Dowelltown Member	30.4	10	60	30	--	--	10YR4/.9
" " " "	37.0	10	55	35	--	--	10YR3/.8

Table 2. Clay mineral ratios of Devonian Shale samples from 2 drill holes in Kentucky

Clay Mineral Ratio

Stratigraphic Unit	Footage	Chlorite	Illite	Swelling Mixed-layer	Non-swelling Mixed-layer	Kaolinite	Color
API No.	16-159-31020		Martin Co., Kentucky				
Ohio Shale - Cleveland Member	2440.0	10	60	10	15	5	10YR3/.8
" " " "	2460.0	10	55	25	5	5	10YR4/.2
" " " "	2480.0	10	55	25	5	5	10YR4/.3
" " " "	2500.0	10	60	20	tr	10	10YR4/.3
" " " "	2520.8	10	55	20	5	10	N5.2
" " " "	2540.2	10	55	20	tr	15	N6.5
" " " "	2560.1	10	55	25	--	10	N6.2
" " " "	2580.3	10	50	25	5	10	N5.2
" " Three Lick Bed	2600.0	5	50	30	10	5	10YR4/.3
" " " " "	2620.0	10	50	20	10	10	N6.7
" " " " "	2639.6	15	50	20	tr	15	N6.7
" " Huron Member, Upper part	2660.0	10	50	25	10	5	10YR4/.3
" " " " " " "	2680.0	10	60	15	10	5	10YR4/.4
" " " " " " "	2700.2	5	60	25	5	5	10YR4/.5
" " " " " " "	2719.5	10	65	20	tr	5	10YR4/.5
" " " " " " "	2740.4	10	60	25	--	5	10YR4/.5
" " " " " " "	2760.0	10	65	20	--	5	10YR4/.1
" " Huron Member, Middle part	2780.9	15	60	25	--	--	N6.5
" " " " " " "	2800.0	20	60	20	--	--	N6.4
" " " " " " "	2820	20	60	20	--	--	N5.0
" " " " " " "	2840	20	50	25	5	--	N5.3
" " " " " " "	2860.0	15	50	25	10	--	10YR5/.3
" " " " " " "	2880.0	15	55	25	5	--	N6.5
" " Huron Member, Lower part	2900.0	20	50	20	10	--	N5.0
" " " " " " "	2920.0	15	55	20	10	--	10YR4/.4
" " " " " " "	2940.8	10	60	25	5	--	N6.2
" " " " " " "	2959.6	10	55	30	5	--	10YR3/.4
" " " " " " "	2980.0	20	65	10	5	--	10YR4/.3
" " " " " " "	3000.0	5	60	20	15	--	N5.5
" " " " " " "	3020.4	10	55	30	5	--	N6.9
" " " " " " "	3040.0	10	75	5	10	--	10YR4/.3
" " " " " " "	3060.0	10	60	15	15	--	10YR4/.5
" " " " " " "	3080.0	15	60	20	5	--	10YR6/.4
" " " " " " "	3100	15	55	20	10	--	10YR4/.5
Java Formation	3120.0	15	55	25	5	--	N6.5
" "	3140.0	20	60	20	--	--	N6.7
" "	3160	20	60	20	--	--	10YR4/.4
" " Pipe Creek Shale Member	3180.0	15	55	20	10	--	N6.8
" " " " "	3200	15	55	25	5	--	N6.8
West Falls Formation	3220.8	20	55	20	5	--	N6.8
" " "	3240.0	20	60	20	--	--	N5.3
" " "	3259.7	20	60	20	--	--	N6.7
" " "	3280.3	15	65	20	--	--	10YR4/.5
" " "	3300.3	15	60	15	10	--	N6.7
" " "	3320.0	15	65	15	5	--	10YR4/.3
" " "	3340.0	15	65	10	10	--	N6.7
" " " Rhinestreet Member	3360.0	10	65	15	10	--	10YR4/.3
" " " " "	3380	10	65	20	5	--	N5.3
" " " " "	3400	10	70	15	5	--	10YR4/.5
API No.	16-193-28982		Perry Co., Kentucky				
Ohio Shale - Cleveland Member	2371.1	15	55	15	15	--	10YR3/.2
" " " "	2381	10	65	15	10	--	10YR3/.2
" " " "	2392.0	10	55	20	15	--	10YR4/.2
" " " "	2404.2	5	60	20	15	--	10YR4/.2
" " " "	2412.0	5	60	15	20	--	10YR4/.2
" " " "	2422.3	10	65	15	10	--	N4.8
" " " "	2432.7	10	60	25	5	--	N4.8
" " Three Lick Bed	2440.9	10	65	20	5	--	N4.3
" " Huron Member, Upper part	2450.7	5	70	20	5	--	N6.5
" " " " " " "	2459.9	10	50	25	15	--	N4.0
" " " " " " "	2470.2	10	65	25	--	--	N4.0
" " " " " " "	2480	5	70	20	5	--	N4.5
" " " " " " "	2491.1	5	65	25	5	--	N4.0
" " Huron Member, Middle part	2501	5	65	20	10	--	N4.5
" " " " " " "	2511.0	5	65	15	15	--	N4.5
" " " " " " "	2519.9	5	70	10	15	--	N4.5
" " " " " " "	2530.3	5	60	35	--	--	N4.5
" " " " " " "	2540	5	65	20	10	--	N4.0
" " " " " " "	2552.4	5	65	15	15	--	N4.0
" " " " " " "	2560	5	65	15	15	--	N4.3
" " " " " " "	2570.0	10	65	15	10	--	N4.2
" " " " " " "	2580	5	65	10	20	--	N4.2
" " Huron Member, Lower part	2590.4	5	65	15	15	--	N4.0
" " " " " " "	2600.2	--	70	25	5	--	10YR4/.2
" " " " " " "	2610.9	5	60	20	15	--	10YR4/.2
" " " " " " "	2621.3	10	60	15	15	--	N6.5
" " " " " " "	2630	10	70	20	tr	--	N6.5
" " " " " " "	2640	5	75	15	5	--	10YR4/.2
" " " " " " "	2650.8	5	65	25	5	--	10YR4/.2
" " " " " " "	2660	15	65	10	10	--	10YR4/.2
West Falls Formation-Olentangy Shale	2671.8	20	70	5	5	--	N6.5
Rhinestreet Shale Member	2680.8	15	65	10	10	--	N6.5
" " " " "	2690	20	55	20	5	--	N6.5
" " " " "	2700	20	60	20	tr	--	N6.5

Table 3. Clay mineral ratios of Devonian shale from a drill hole in Virginia

Clay Mineral Ratio

Stratigraphic Unit	Footage	Chlorite	Illite	Swelling Mixed-layer	Non-swelling Mixed-layer	Kaolinite	Color
API No.	45-195-20253		Wise Co., Virginia				
Ohio Shale - Cleveland Member	4870.6	15	55	25	--	5	N3.3
" " " "	4875.9	10	60	25	5	--	N3.5
" " " "	4880.0	10	60	30	tr	--	N3.3
" " " "	4885.1	10	55	35	--	--	N3.2
" " " "	4890.1	10	60	30	--	--	N3.2
" " " "	4895.5	5	55	40	--	--	N3.4
" " " "	4900.0	5	55	40	--	--	N3.2
" " " "	4905.4	5	75	20	--	--	N3.2
" " " "	4910.1	5	55	40	--	--	N3.2
" " " "	4915.0	5	65	30	--	--	N3.2
" " " "	4920.2	5	65	30	--	--	N3.5
" " " "	4925.2	10	60	25	5	--	N3.5
" " " "	4930.2	10	50	25	10	5	N3.5
" " " "	4935.0	10	60	25	--	5	N3.5
" " " "	4940.2	10	60	25	--	5	N3.5
" " Three Lick Bed	4945.1	10	70	15	--	5	N4.0
" " " "	4950.8	10	55	25	--	10	N4.0
" " " "	4955.3	10	60	25	--	5	N5.0
" " " "	4960.1	10	55	30	--	5	N3.7
" " " "	4965	20	50	25	--	5	N6.8
" " " "	4970	15	65	20	--	--	N6.0
" " " "	4974.2	20	55	25	--	--	N6.0
" " " "	4980.0	15	60	25	--	--	N6.0
" " " "	4985.1	20	60	20	--	--	N4.7
Ohio Shale- Huron Member, Middle part	5210	10	70	20	--	--	N6.0
" " " " " " "	5215	15	55	30	--	--	N5.3
" " " " " " "	5220	15	60	25	--	--	N6.2
" " " " " " "	5225	10	65	25	--	--	N5.7
" " " " " " "	5230	15	60	25	--	--	N6.2
" " " " " " "	5235.0	15	55	30	--	--	N5.7
" " " " " " "	5240.2	15	65	20	--	--	N4.7
" " " " " " "	5244.9	10	60	30	--	--	N4.7
" " " " " " "	5250	10	60	30	--	--	N3.9
" " " " " " "	5255	15	60	25	--	--	N5.0
" " " " " " "	5260.0	10	60	30	--	--	N4.7
" " " " " " "	5265.0	15	55	30	--	--	N3.9
" " " " " " "	5270.1	10	60	30	--	--	N5.0
" " " " " " "	5275.1	10	65	25	--	--	N4.0
" " " " " " "	5280.0	15	60	25	--	--	N5.7
" " " " " " "	5285	10	65	25	--	--	N4.1
" " " " " " "	5290.0	15	65	20	--	--	N4.1
" " " " " " "	5295.6	15	65	20	--	--	N4.1
Ohio Shale- Huron Member, Lower part	5300.7	15	80	5	--	--	N4.0
" " " " " " "	5305.2	10	55	35	--	--	N3.7
" " " " " " "	5312	15	50	35	--	--	N3.7
" " " " " " "	5315.9	10	65	25	--	--	N3.7
" " " " " " "	5320.0	10	60	30	--	--	N3.7
" " " " " " "	5325	10	60	30	--	--	N4.0
" " " " " " "	5330.3	10	60	25	5	--	N4.0
" " " " " " "	5340.0	15	55	25	5	--	N4.0
" " " " " " "	5345.8	15	60	25	--	--	N3.5
" " " " " " "	5350.0	10	70	20	--	--	N3.5
" " " " " " "	5355	10	60	30	--	--	N5.7
" " " " " " "	5360	5	70	25	--	--	N3.5
" " " " " " "	5365.0	10	65	25	--	--	N3.2
" " " " " " "	5370.7	10	70	20	--	--	N3.2
" " " " " " "	5375.2	10	60	30	--	--	N3.2
" " " " " " "	5379.9	10	70	20	--	--	N3.2
" " " " " " "	5385.0	10	60	30	--	--	N3.2
" " " " " " "	5390	10	65	25	--	--	N3.2
" " " " " " "	5395.5	10	65	25	--	--	N6.0
" " " " " " "	5400	10	65	25	--	--	N3.4
" " " " " " "	5405.4	10	65	25	--	--	N3.4
" " " " " " "	5409.8	10	65	20	5	--	N3.2
" " " " " " "	5415.4	10	65	25	--	--	N3.2
" " " " " " "	5420.3	10	60	25	5	--	N3.2
" " " " " " "	5425.0	10	70	20	--	--	N3.5
" " " " " " "	5430.4	10	60	30	--	--	N3.2
" " " " " " "	5435	10	70	20	--	--	N3.4
" " " " " " "	5440	15	60	25	--	--	N4.1
" " " " " " "	5445	10	65	25	--	--	N3.5
" " " " " " "	5450.0	15	65	15	5	--	N4.1
" " " " " " "	5456.8	10	60	25	5	--	N3.2
" " " " " " "	5460.6	10	65	20	5	--	N3.2
" " " " " " "	5465.2	10	65	20	5	--	N3.2
" " " " " " "	5470.9	10	65	20	5	--	N3.2
" " " " " " "	5475.2	10	65	20	5	--	N3.2

Table 5. Clay mineral ratios of Devonian shale samples from 2 drill holes in Ohio

Clay Mineral Ratio

Stratigraphic Unit	Footage	Chlorite	Illite	Swelling Mixed-layer	Non-swelling Mixed-layer	Kaolinite	Color
API No.	34-019-20835			Carroll Cos., Ohio			
Ohio Shale-Huron Member, Upper part	2080.2	25	50	10	5	10	N7.0
" " " " " "	2090.8	20	40	15	15	10	N6.0
" " " " " "	2100.0	20	50	15	--	15	[illegible]
" " " " " "	2110	25	45	20	--	10	N6.2
" " " " " "	2120.0	20	45	15	15	5	N6.2
" " " " " "	2130.0	20	40	20	tr	20	N7.0
" " " " " "	2140.0	20	45	20	5	10	N6.2
" " " " " "	2150.0	20	40	20	10	10	N6.1
" " " " " "	2160.5	15	50	20	5	10	N6.3
" " " " " "	2170.0	20	50	15	10	5	N6.2
" " " " " "	2180.0	15	55	20	5	5	N6.0
" " " " " "	2190.0	20	50	25	5	--	N6.2
" " " " " "	2200.0	15	50	30	5	--	10YR4/.2
West Falls Formation-Rhinestreet Shale Member	3080.3	10	65	15	10	--	N3.8
" " " " "	3090.4	20	60	20	tr	--	N6.2
" " " " "	3100	15	70	5	10	--	N3.8
" " " " "	3111.0	10	60	15	15	--	10YR4/.2
" " " " "	3120.0	10	60	25	5	--	10YR3/.2
" " " " "	3130.0	15	55	20	10	--	10YR4/.2
" " " " "	3139.0	15	70	10	5	--	10YR4/.2
" " " " "	3150.0	15	65	15	5	--	10YR4/.2
Olentangy Shale	3160.0	20	65	10	5	--	N6.0
" "	3170.0	25	55	20	tr	--	N6.0
" "	3180.8	30	50	15	5	--	N6.1
" "	3189.6	30	45	15	10	--	N6.0
API No.	34-167-23521			Washington Co., Ohio			
Ohio Shale- Huron Member, Middle part	3490	20	60	10	5	5	N6.5
" " " " " " "	3500.1	15	50	10	5	20	N7.3
" " Huron Member, Lower part	3510.4	15	55	15	5	10	N4.1
" " " " " " "	3520.0	20	55	10	10	5	N4.3
" " " " " " "	3525	15	55	15	10	5	N6.0
" " " " " " "	3530.0	15	50	25	10	--	N4.3
" " " " " " "	3532.3	20	60	15	5	--	N4.0
" " " " " " "	3540.3	20	55	15	10	--	N4.0
" " " " " " "	3550.1	20	50	25	5	--	N6.0
" " " " " " "	3559.6	20	50	25	5	--	N3.5
" " " " " " "	3570.9	15	50	30	5	--	N4.5
" " " " " " "	3580.7	10	55	20	15	--	N4.0
" " " " " " "	3590	15	50	20	15	--	N4.5
" " " " " " "	3600.5	15	50	25	10	--	N4.5
" " " " " " "	3609.0	20	50	30	--	--	N4.0
" " " " " " "	3620.6	20	40	25	15	--	N6.0
" " " " " " "	3630.4	20	45	30	--	5	N4.0
" " " " " " "	3640.9	20	55	15	--	10	N6.0
" " " " " " "	3650	15	60	20	--	5	N5.6
" " " " " " "	3660.9	20	50	20	5	5	N6.2
" " " " " " "	3670	15	55	15	15	--	N4.3
" " " " " " "	3680	15	55	15	10	5	N6.3
" " " " " " "	3690	10	55	20	5	10	10YR6/.2
" " " " " " "	3700.4	10	60	30	--	--	N3.8
" " " " " " "	3710	15	50	15	10	10	N6.0

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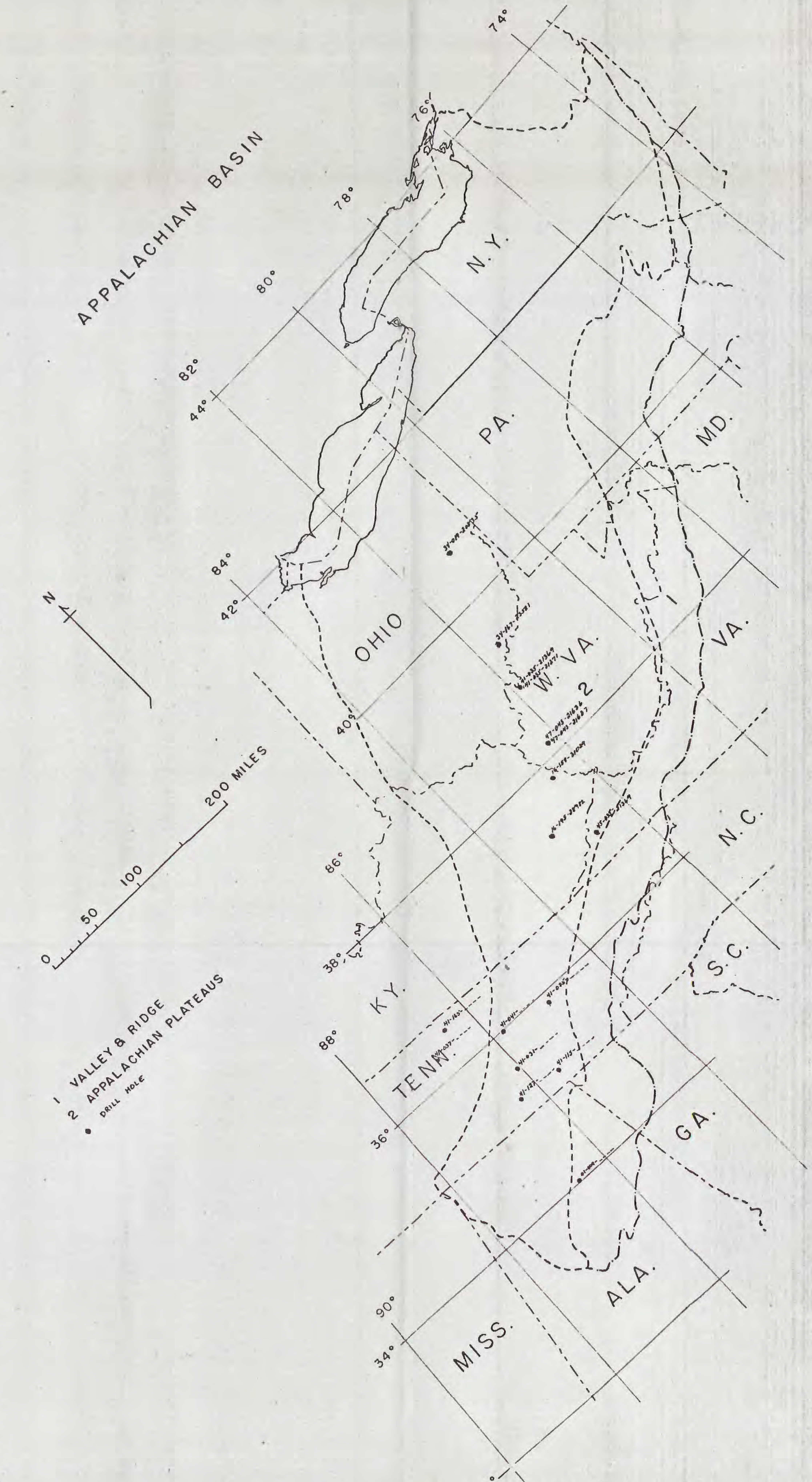

Figure 1. Index map showing location of drill holes

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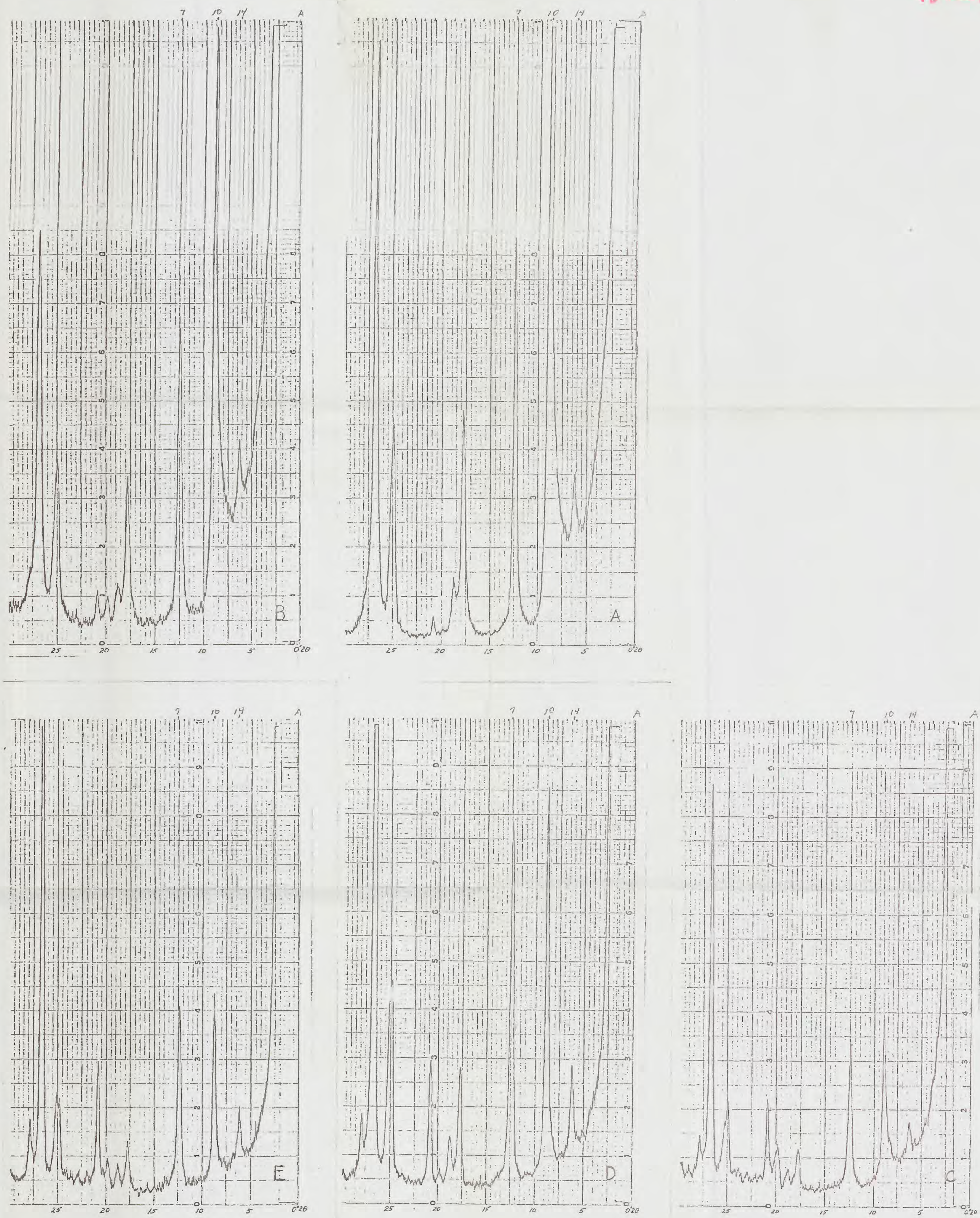

FIGURE 2

78-1084

Table 4. Clay mineral ratio of Devonian shale samples from 4 drill holes in West Virginia

Stratigraphic Unit	Footage	Clay Mineral Ratio					Color
		Chlorite	Illite	Swelling Mixed-layer	Non-swelling Mixed-layer	Kaolinite	
API No.	47-035-21369		Jackson Co., West Virginia				
Ohio Shale-Huron Member, Middle part	3410	20	55	25	tr	--	N4.1
"	3420.6	15	60	25	--	--	N5.1
"	3430	10	55	30	--	5	N5.0
"	3439.1	15	50	25	5	5	N4.5
"	3450.4	15	45	20	10	10	N5.0
"	3470	15	55	25	tr	5	N4.3
"	3480	10	55	25	--	10	N4.7
"	3489.0	15	45	15	5	20	N7.0
"	3497.8	15	45	15	10	15	N7.0
Ohio Shale- "	3622.0	20	55	20	5	--	N6.5
"	3630	20	55	25	--	--	N5.0
"	3640	15	50	20	5	15	N6.5
"	3644	10	50	15	5	20	N7.0
"	3654.3	20	60	20	--	--	N4.0
Ohio Shale-Huron Member, Lower part	3660.6	15	65	20	--	--	N4.0
"	3680.5	20	60	20	--	--	N4.0
"	3690	20	45	25	10	--	N4.0
"	3700	20	65	15	tr	--	N3.8
"	3720	20	55	25	--	--	N3.8
"	3730	20	55	25	--	--	N6.7
"	3740	20	70	10	--	--	N3.9
"	3750	20	60	20	--	--	N3.9
"	3759.4	15	55	30	--	--	N6.9
"	3770	15	60	15	10	--	N6.5
"	3780	20	60	15	5	--	N4.0
"	3790	20	55	20	5	--	N6.5
API No.	47-035-21371		Jackson Co., West Virginia				
Ohio Shale-Huron Member, Middle part	3220	15	50	30	5	--	N4.5
"	3230	20	50	25	5	--	N6.0
"	3240	15	60	15	10	--	N4.5
"	3250	15	55	10	tr	20	N6.0
"	3260	20	55	10	5	10	N4.5
"	3270	25	55	20	--	--	N6.5
"	3280	20	55	25	--	--	N4.8
"	3290	15	55	20	--	10	N6.5
"	3300	20	55	15	--	10	N4.3
"	3310	20	50	20	--	10	N5.0
"	3320	15	55	20	--	10	N6.5
"	3330	15	55	20	--	10	N6.5
"	3340	15	50	15	10	10	N5.5
"	3350	15	50	20	5	10	N6.5
"	3360	15	50	20	10	5	N5.5
"	3380	25	45	20	10	--	N6.2
"	3390	25	45	25	5	--	N6.5
"	3400	25	50	15	10	--	N5.3
"	3410	25	50	25	tr	--	N6.7
"	3420	25	50	20	5	--	N6.7
"	3440	25	50	25	tr	--	N6.7
"	3450	25	50	25	--	--	10YR5/.3
Ohio Shale-Huron Member, Lower part	3460	20	55	25	--	--	10YR5/.3
"	3470	20	50	30	--	--	10YR4/.3
"	3480	25	45	30	--	--	N6.7
"	3490	15	55	25	5	--	10YR4/.3
"	3500	15	65	15	5	--	10YR4/.3
"	3510	20	55	20	5	--	10YR4/.3
"	3520	20	55	15	10	--	10YR4/.3
"	3530	20	50	25	5	--	N6.6
"	3540	20	55	15	10	--	10YR4/.3
"	3550	15	55	15	15	--	10YR4/.3
"	3560	15	50	20	15	--	10YR4/.3
"	3570	20	55	10	15	--	10YR4/.3
"	3580	20	55	10	15	--	10YR4/.3
"	3590	15	50	15	20	--	10YR4/.3
"	3600	15	55	25	5	--	10YR4/.3
"	3610	20	50	15	15	--	N6.7
"	3620	15	65	15	5	--	N5.6
"	3640	15	55	15	15	--	10YR4/.3
Java Formation	3650	20	45	20	15	--	N4.3
"	3660	25	50	20	5	--	N6.7
"	3670	20	55	15	10	--	N6.7
"	3680	30	40	25	5	--	N6.8
"	3690	25	50	15	10	--	10YR4/.3
	4020	15	70	10	5	--	10YR4/.3
	4030	15	75	5	5	--	10YR4/.3
API No.	47-043-21639		Lincoln Co., West Virginia				
Ohio Shale-Chagrin Member	2660	15	50	20	5	10	N5.7
"	2670	15	50	15	10	10	N6.2
"	2680	20	55	5	5	15	N6.7
"	2690.9	25	45	10	5	15	N6.7
"	2700.8	20	40	20	5	15	N6.2
"	2709.9	20	50	10	5	15	N6.7
"	2720	20	55	10	5	10	N5.7
"	2730	20	50	15	tr	15	N6.8
"	2740	25	45	15	tr	15	N6.8
"	2750.6	20	45	20	--	15	N6.8
"	2760	15	70	15	--	--	N3.0
"	2770	15	60	15	tr	10	N5.7
Ohio Shale-Huron Member, Middle part	3000	15	45	20	5	15	N5.7
"	3010	15	45	25	tr	15	10YR5/.2
"	3020.8	15	50	15	5	15	N6.5
"	3030	15	55	20	--	10	10YR5/.2
"	3040.3	10	55	25	--	10	N5.3
"	3050.5	10	55	25	tr	10	N5.3
"	3059.7	15	50	25	--	10	N5.3
"	3070	15	45	20	10	10	10YR5/.2
"	3080	15	50	20	5	10	N5.3
"	3090	15	60	10	5	10	N6.7
"	3100	15	55	15	5	10	N6.7
"	3110	10	50	25	5	10	N6.5
Ohio Shale-Huron Member, Lower part	3290.9	15	50	20	5	10	N6.5
"	3300	15	50	20	5	10	10YR5/.2
"	3320	[illegible]	50	25	--	10	N6.5
"	3330	15	55	15	10	5	N5.7
"	3340	15	60	15	tr	10	10YR5/.2
"	3350	20	55	10	15	--	10YR4/.2
"	3360	20	60	10	10	--	10YR4/.2
"	3370	15	60	15	10	--	10YR4/.2
"	3380	15	60	20	5	--	N5.7
"	3390	20	55	15	10	--	10YR4/.3
"	3399.0	15	55	20	10	--	N5.5
"	3409	20	75	5	--	--	10YR4/.3
"	3420.1	15	70	10	5	--	10YR4/.3
"	3439.9	15	65	10	10	--	10YR4/.3
"	3450.5	20	60	20	tr	--	10YR4/.3
"	3460	10	80	10	--	--	10YR4/.3
"	3470	15	70	10	5	--	N6.1
"	3480	10	70	10	10	--	N5.7
"	3489.4	10	70	10	10	--	10YR4/.3
"	3499.6	15	55	20	10	--	10YR4/.3
"	3509.8	10	70	15	5	--	10YR4/.3
"	3520	10	65	10	15	--	10YR4/.3
"	3530.6	15	70	5	10	--	10YR4/.3
"	3550	15	60	5	20	--	N5.7
"	3560.6	20	60	tr	30	--	10YR4/.3
"	3569.8	15	60	15	10	--	10YR4/.3
"	3579.7	15	75	5	5	--	10YR4/.3
West Falls Formation-Rhinestreet Shale Member	3889.8	20	60	10	10	--	10YR4/.3
"	3899.3	10	65	15	10	--	10YR4/.3
"	3909	10	70	15	5	--	10YR4/.3
"	3920	10	70	10	10	--	N5.3
"	3929.7	15	65	10	10	--	10YR4/.3
"	3940	15	55	20	10	--	10YR4/.3
"	3950	20	60	10	tr	10	N5.7
"	3960	20	80	--	--	--	10YR4/.3
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Ohio Shale-Huron Member, Middle part	2725	35	30	10	5	20	N6.8
"	2750	15	55	15	5	10	N6.2
"	2775.9	15	50	20	tr	15	N5.9
"	2799	25	45	5	10	15	N6.8
"	2824	25	50	10	--	15	N6.8
"	2854.5	15	55	15	tr	15	N6.3
"	2875	15	60	10	--	15	N6.2
"	2900	15	50	25	5	5	N5.8
"	2925	15	55	20	--	10	N5.5
"	2950	10	55	25	--	10	N5.7
"	2975	10	60	20	--	10	10YR4/.3
"	3001	10	55	25	--	10	N5.5
"	3024.9	10	50	30	tr	10	N5.7
"	3050	10	55	25	--	10	N5.7
"	3075	10	50	20	10	10	N6.0
"	3100	15	50	30	--	5	N5.3
"	3125.1	10	50	30	tr	10	N5.3
"	3149	10	55	25	--	10	N5.3
"	3174.9	15	45	30	--	10	N5.3
"	3201.5	10	50	30	--	10	N6.0
"	3225	15	45	30	tr	10	N5.3
"	3249	15	45	30	--	10	N6.0
"	3275	15	55	20	--	10	N6.0
"	3300	15	55	20	--	10	N6.0
"	3325	15	50	25	--	10	N5.5
"	3351.5	15	50	30	--	5	N5.5
"	3374.9	15	65	20	--	--	N5.3
"	3400	20	55	25	--	--	N4.1
Ohio Shale-Huron Member, Lower part	3425	15	60	25	--	--	N6.0
"	3450	15	60	25	--	--	N6.0
"	3475	20	60	20	--	--	N6.0
"	3505	15	55	30	--	--	10YR4/.3
"	3525	15	60	15	10	--	10YR4/.5
"	3550	15	60	20	5	--	10YR4/.5
"	3575	15	60	20	5	--	10YR4/.5
"	3600	15	55	30	--	--	10YR4/.5
"	3625	15	65	20	--	--	10YR4/.5
"	3652	20	50	30	--	--	N6.6
Java Formation	3675	15	55	30	--	--	N6.3
"	3700.5	20	55	25	--	--	N6.9
"	3725.6	25	50	25	--	--	N6.5
"	3750	15	55	30	--	--	N6.5
"	3774.5	15	65	20	--	--	N6.5
"	3799.5	20	55	20	5	--	N6.5
"	3825	25	55	20	tr	--	N5.6
"	3850	20	60	15	5	--	N6.6
West Falls Formation-Rhinestreet Shale Member	3875.3	20	55	15	10	--	N6.3
"	3900	20	60	15	5	--	N6.3
"	3925	20	55	15	10	--	N5.9
"	3949	15	70	15	--	--	10YR4/.5
"	3974	10	70	20	--	--	10YR4/.5
"	4000	15	65	20	--	--	10YR4/.2
"	4025	15	70	15	--	--	10YR4/.5